Experimental and numerical multiscale approach to thermally cycled FRP

Caroline Lüders^{a,b*}, Gerhard Kalinka^{c*}, Wei Li^c, Michael Sinapius^{a,b}, Tobias Wille^b

^aTechnische Universität Braunschweig, Langer Kamp 6, 38106 Braunschweig ^bGerman Aerospace Center, Lilienthalplatz 7, 38108 Braunschweig ^c Bundesanstalt für Materialforschung und -prüfung, Unter den Eichen 87, 12205 Berlin

*Corresponding author.

E-Mail address: c.lueders@tu-braunschweig.de, caroline.lueders@dlr.de (C. Lüders), gerhard.kalinka@bam.de (G. Kalinka)

Abstract

5

10

15

20

25

Due to the different thermal expansion of the constituent materials, cyclic thermal loading of FRP induces alternating stresses in the material at two scales: at the micro scale (level of fibre–matrix-interaction) and at the macro scale (level of the multidirectional laminate). Especially the micro scale effect is not comprehensively investigated yet. Additionally, computational investigations mostly neglect this effect due to the homogenous modelling of the composite material. As this effect is assumed to significantly contribute to the fatigue of FRP at thermal loads, the present paper suggests an experimental and numerical multiscale approach including experiments at the different involved material scales to separately observe the effects acting at these scales. The approach also includes numerical modelling for each scale to complement the knowledge gained from the experiments and to create a basis for the consideration of the micro effect even in macroscopic fatigue models treating homogeneous modelled composites. The main focus of the contribution is to bring the overall approach up for discussion, rather than to present the multiscale modelling details.

Keywords: Fatigue, thermal cycling, fibre-reinforced plastics, microscale, macroscale, cryogenic, carbon fibre, epoxy resin

1. Introduction

35

40

45

50

55

60

Due to their superior mechanical properties in conjunction with their low density fibre reinforced plastics (FRP) are increasingly used as a material for various lightweight constructions. For example, focussing on alternative fuel concepts in the automotive industry, hydrogen storage tanks made of carbon fibre reinforced plastics (CFRP) are discussed [1]. Pressure vessels storing cryo-compressed hydrogen (CcH2) are exposed to not only mechanical loads due to the internal pressure of 350 MPa, but are also thermally loaded due to the cold hydrogen (33.15 K). As the process of refuelling and discharge is repeated during the operational life, the material is exposed to cyclical thermal and mechanical loading. Assuming an average use of 15 years, the pressure vessel has to sustain about 1000 of these thermo-mechanical load cycles [1]. Other examples for structural elements which are cyclically thermal loaded are satellites which are exposed to varying solar radiation. And for off-shore civil engineering and for aerospace applications the influence of freeze-thaw cycles on the mechanical performance is investigated [2][3].

In FRP, cyclically thermal loads induce alternating stresses at different scales [4] (ref. Figure 1). At the fibre-matrix-scale (micro level) stresses are induced in the fibres, matrix and in the fibre-matrix-interface due to the different thermal expansion of fibre and matrix. These differences between the composite constituents yield to an anisotropic thermal expansion behaviour of a unidirectional (UD) composite ply. In thermally loaded multidirectional (MD) laminates (macro level), made of several UD plies with different fibre orientation, this leads to thermally induced stresses inside the single plies and in the interfaces between the plies. As for mechanical cyclic loads [5][6], the damage initiation at thermal cycling is observed at the fibre-matrixinterface [7][8][9]. Microscopic techniques are used to observe the initiation and growth of these debondings. But investigations beyond microscopy for investigating the interface, for example single fibre-pull-out tests [9][11][17][18] after thermal cycling, are not yet present in the literature. The most experimentally investigations on the fatigue of FRP at thermal loads focus on MD laminates [7][8][19][20][21] in which the influence of the micro and macro effect cannot be separated. Only few researchers investigate UD laminates and the micro level effect [22][23][24][26][29].

The findings of these works partly diverge and are incomplete. E.g., often only individual material parameters are measured after thermal cycling. Preliminary the properties in fibre direction are investigated and no significant degradation of these properties is observed [24][26]. Only a few researches examine matrix dominated properties, which are limited to bending and transverse tension properties [24][29]. The current findings of research cannot explain the fundamental issues of the influence of thermal cycling on the mechanical properties. Therefore a comprehensive mechanical material characterisation is required after cyclical thermal conditioning. An attempt in this direction is part of the presented research program and is published by the authors [30]. Computational investigations - using analytical models or Finite Element Method (FEM) - of thermal loaded FRP are mostly done on the UD ply level whereby the composites are considered to be homogeneous [20][31][32][33][34][35][36]. For the assessment of cyclic thermal loads the modelling approaches for mechanical fatigue are transferred [31][37][38][39]. Due to the homogeneous modelling of the UD ply, thermally induced stresses on the micro scale cannot be considered. This disadvantage could be overcome by a multiscale approach as presented in the literature for static thermal loading [40][41][42]. In these approaches, the damage calculation is based on micro level stresses. Failure of the composite constituents leads to degradation of the macroscopic UD ply properties. This way, thermally induced stresses on microscale are considered. Currently, the application of these approaches is limited to thermal static loading. Additionally, the calculation of the micro level stresses form the macro level stresses assumes linearity of the material behaviour whereby not even the temperature dependency of the material properties can be considered. But the computation time for a fully coupled multiscale simulation including a top-down and a bottom-up scale transfer to consider nonlinear effects is quite high. This may be acceptable for a static analysis, but will not be manageable considering cyclic effects. In summary, for a profound understanding and reliable simulation of the damage mechanisms in FRP under cyclic thermal loads, more clearly focused experiments are necessary as well as respective validated simulation models. This paper presents an experimental and numerical concept that wants to achieve this.

65

70

75

80

85

2. Leading hypotheses

95

100

105

110

115

120

The central hypothesis of the present research is that intra-ply stresses at the micro level and inter-ply stresses at the macro level, that are caused by the different thermal expansion of fibre and matrix, are responsible for damage and fatigue of cyclic thermal loaded FRP. While in a thermally cycled UD laminate only the first micro level effect is present, in a MD laminate both effects are superimposed. It is assumed, that already the intra-ply stresses (micro effect) alone causes significant fatigue damage, because - this is the assumption - the alternating thermally induced stresses at the fibre-matrix-interface cause a weakening of this interface. As the intra-ply stresses are assumed to significantly contribute to fatigue damage, they should be considered within thermal fatigue calculations. This is not possible, if the composite ply is modelled as homogeneous medium. On the other hand, computationally intensive multiscale approaches seem not to be expedient for fatigue assessment of large structures. That is why a bottom-up-approach is proposed, ref. Figure 2. This approach assumes the existence of an equivalent mechanical stress state of the UD ply which leads to the same damage as the thermally induced stresses at micro scale. For fatigue simulation of a thermally loaded MD composite modelled by homogeneous single plies, first, the macroscopic thermally induced stresses are calculated for each UD ply. Then, for each ply, the mechanical stress state corresponding to the damage caused by the thermally induced stress at micro scale is derived. This could be done using analytical meta-models created from micromechanical simulations in advance. This equivalent stress state is superimposed to the macroscopic ply stresses. These superimposed stress states are then together assessed by a macroscopic fatigue damage model predicting a degradation of the single ply properties. The equivalent stress state contains all nonlinearities considered in the micromechanical model and transfers their macromechanical effects to the macro scale. The existence of such an equivalent mechanical stress state expects the same fatigue damage mechanisms at mechanical and at thermal cyclic loads. It is assumed, that the observed differing crack patterns and crack progression results from the different inner stress state caused by mechanical and thermal loads. To summarize, the leading hypotheses of the

present study are:

125

130

145

- 1. Alternating thermally induced stresses at the micro scale cause damage of the fibre-matrix-interface.
- 2. The contribution of thermal induced stresses on the micro scale to the fatigue of FRP under thermal loads is significant.
- The fatigue damage mechanisms are the same at mechanical and thermal loading. The differing crack patterns and crack propagation is caused by the different inner stress state.
- The thermally induced stress at micro scale can be considered in macroscopic fatigue damage models by a damage equivalent mechanical stress state.
- For investigating these hypotheses, experimental testing and modelling is required across the material scales. The considered, separable material scales are depicted in Figure 1. The smallest scale is the micro scale of the fibre-matrix-level. On this scale it is looked at two different model sizes. The first is the fibre-matrix-interaction at the interface. Here, the interface of one single fibre is investigated. The second micro scale model consist of many fibres for analysing the debonding of additional interfaces and their coalescence forming matrix cracks in UD composites. The multiscale approach ends up on the macro scale represented by MD composites.
 - With respect to the use case of CcH2 pressure vessels [1], the temperature and cycle range of the study is chosen to be 1000 thermal cycles between room temperature (293 K) and 90K. This minimum temperature can be realized for specimens using liquid nitrogen (LN2) as cooling agent and not allowing contact between the LN2 and the specimen for preventing thermal shock. Realizing the minimal service temperature of CcH2 pressure vessels of 33.15 K would require the use of helium or hydrogen as cooling agent which is associated with very high costs or high elaborate safety precautions, respectively. If matrix cracks are caused by 1000 thermal cycles between room temperature and 90K, the phenomena could nevertheless be investigated. The investigated material is a carbon fibre reinforced plastic (CFRP) made of IM7 fibre and an epoxy resin. The specific experimental setups at the different scales and the

main idea of the modelling approaches used to get a deeper understanding of the observed phenomena are described in the next sections starting at the smallest scale. The authors would like to point out, that the focus of this contribution is on the main concept for investigating the micro and macro effect in thermally cycled FRP, which is brought up for discussion. Detailed information about the respective modelling approaches and detailed simulation or experimental results are subject of already published [30][56] or planned publications. Nevertheless, for some experiments and simulations some results are presented in a summarised manner. After discussing the phenomena and the models of the different scales, the scale transfer approach for the multiscale analysis is explained in more detail. In the last section of the paper, a summary of the presented experimental and numerical approach is given, some expectations of the experimental results a derived and a perspective of the numerical predictability is given.

3. Micro scale – level of fibre-matrix-interaction

Stresses on the micro level arise between single fibres and the surrounding matrix material. These could be combinations between shear stresses and radial stresses normal to the fibre's surface. As a consequence, thermal cycling may cause decohesion in some area of contacts between fibre and matrix resulting in a weakening of the interface properties. There are several mechanical tests available to test fibre reinforced composites: for example a variation of tensile tests (e.g. applying in-plane or out-of-plane stresses regarding the fibre orientation), shear tests or lap shear tests, and short beam tests for the determination of the interfacial shear strength (ILSS). However, all these tests address the complete combination of fibres, matrix and interfaces between the constituents. Consequently, the results are influenced by a widely unknown combination of fibre properties (e.g. variation of the local volume fraction, local fibre orientation), the properties and distribution of the matrix material (e.g. cracks in the matrix), and the bonding conditions between fibres and the matrix. Therefore, the integral results of such tests could not be traced back to the interface properties only. That is the motivation for using "micromechanical" test methods as

the single fibre pull-out test [10][11], the single fibre push-out test [12] or the single fibre peel-off test [38]. These tests address the fibre-matrix interface directly. The load introduced in a single fibre can be precisely controlled. This load is transferred by shear stresses through the fibre-matrix interface in the surrounding material. Thus, all phenomena affecting the fibre-matrix bond strength lead directly to an observable change of the interfacial shear strength. Other known micromechanical test methods are the micro droplet test [13][14] and the fragmentation test [15][16]. During the micro droplet test an inhomogeneous stress field along the droplet is introduced and the interfacial stresses are affected by the droplet geometry. In the fragmentation test, a soft matrix material is required to reach a saturation of the fragmentation which is necessary for evaluation. Additionally, the evaluation method requires assumptions which cannot easily be validated. Due to these shortcomings the pull-out and the peeloff tests are preferred for measuring the strength of the fibre-matrix-interface after thermal cycling. The degrading of this interface strength is assumed to be a function of the number and the intensity of the thermal cycling. For investigating this degradation, single fibre samples have to be thermally cycled and the pull-out and the peel-off-test have to be performed afterwards at room temperature. By testing samples conditioned by different numbers of thermal cycles, the interface properties can be drawn as function of the number of thermal cycles. Therefore, the samples will be conditioned in a temperature chamber enabling thermal cycling between room temperature and 90 K. In the first step, a conditioning with 1000 cycles will be applied. An increasing of the cycle number is planned until a significant degradation of the interface properties is observed. The pull-out and peel-off tests will be performed after conditioning at room temperature.

185

190

195

200

205

210

3.1 Thermal cycling apparatus for single fibre samples

For the well-defined thermal cycling of single fibre samples, a small-scale temperature chamber has to be developed, ref. Figure 3. It enables a heating/cooling rate of 2K/s. In order to reach this rate, a closed loop control is used involving an electrical heater for temperatures above room temperature and a magnetic valve

releasing small quantities of LN2 for cooling down. In order to ensure a uniform temperature distribution inside the chamber, an air circulation device is installed.

3.2 Design of Pull-Out-Samples

215

220

225

230

235

The conventional design of the pull-out test sample consists basically of a liquid droplet of resin and a fibre which is embedded with a defined length in this droplet. After curing, the fibre will be pulled out of the droplet. Usually, the interfacial shear strength τ is estimated by the relation between the maximal pull-out force F_{max} to the contact area

$$\tau = \frac{F_{max}}{2\pi R_f l_e}$$

where R_f is the radius of the embedded fibre and l_e the embedding length. However, for investigating the impact of thermal cycling on the contact conditions between fibre and matrix according to the first hypothesis, the surrounding of the embedded fibre has to be taken into account and the conventional sample design has to be modified. To emulate the stress state around a fibre within a real composite material, the single fibre is embedded between two platelets of UD composite, where the fibre direction points in the same direction as the single fibre designated for pull-out, as can be seen in Figure 4. The embedding length should be around 5-10 times of the fibre diameter (25-50 μ m). The glue in the gap is the same as for impregnating the composite material. For easier arrangement, the gap is realized as a crack in the composite material.

3.3 Design of Peel-Off-Samples

As is depicted in Figure 5, with the peel-off test only one side of the tested fibre is attached to the substrate. Similar to the sample design for the pull-out test, the thermal expansion of the material surrounding the tested fibre should be as similar as possible to the expansion of the real composite. Therefore, a polished surface of UD composite is used, and the tested fibre is aligned in the same direction as the fibres in the

substrate. A thin layer of resin is used for attaching the testing fibre and the layer is approximately half as thick as the fibre diameter. For this preparation, spin coating is used. The advantage of the peel-off test is that there is no significant influence of friction, since only mode I failure arises. Furthermore, the adhesion work can be easily obtained by integration of the area between peel force and peel length and the relation of this amount of energy to the newly created surface.

4. Micro scale – level of the unidirectional composite

240

245

250

255

260

The second scale is the level where fibre and matrix are considered explicitly but the interaction between several interface damages and the matrix crack forming process is analysed. The investigations at this micro scale are conducted for testing the second hypotheses and to evaluate the influence of the micro scale effect on the fatigue of thermally cycled composites. Therefore, the micro scale investigations are carried out by looking at UD composites.

4.1 Fatigue phenomena in thermally cycled composites at the micro scale

For investigating the fatigue phenomena in thermally cycled composites, UD composite specimens are thermally cycled and tested afterwards under quasi-static mechanical loading at room temperature to obtain the residual elasticity and strength parameters of the material as function of the number of thermal cycles. The expected increasing degradation of the material properties is the macroscopically visible effect of fatigue. The material parameters are determined in longitudinal and transverse fibre direction under tensile and compression loading. To investigate the crack forming process the conditioned specimens are investigated using microscopic techniques. It is expected to observe fibre-matrix-debondings at a certain number of thermal cycles and matrix cracks with increasing crack density at higher cycle numbers. Further details about the experimental setup for the thermal cycling, the mechanical testing,

the microscopic investigation and the strategy result evaluation are already described by the authors [30]. The results of the experiments are briefly summarized: The thermal cycling by 1000 cycles between room temperature and 90 K causes no significant matrix cracking in the UD composite made of the investigated material (IM7/epoxy). Therefore, no significant degradation of the elasticity and strength parameters is observed, too. This falsifies the second hypotheses in the range of the tested cycles, the tested temperature regime and the investigated material. It can be assumed that a higher number of thermal cycles may initiate matrix cracks. Therefore additional microscopy specimens are planned for cycling up to 10 000 thermal cycles using the thermal cycling apparatus for single fibre samples, described above and shown in Figure 3.

265

270

275

280

285

290

4.2 Micromechanical modelling of fatigue phenomena in FRP

A micro scale modelling of fatigue phenomena at fibre-matrix-level allows a deeper insight into the damage process and the damage interaction. The simulation shall also provide a further possibility of comparing the damage mechanism under mechanical and thermal cyclic loading (ref. hypotheses 4). Hence, by the micromechanical modelling two objectives are pursued. The first is to get a deeper understanding of the phenomena on micro scale and the second is to derive an equivalent mechanical stress state for the thermal cyclic loads which is then be used in macromechanical fatigue analyses to capture the micro scale effect.

For modelling the micro scale by FEM the representative volume element (RVE) approach with periodic boundary conditions is used. Building up the RVE requires the fibre diameter ($5.2~\mu m$) and the fibre volume fraction (62%) of the UD composite. First analyses are done using an RVE with hexagonal fibre arrangement, see Figure 6. Its small size (in contrast to RVE with randomised fibre arrangements) facilitates the development of appropriate material models and allows a faster computation of cycle-by-cycle fatigue analyses. Its regular structure also may ease the detection of differences in the damage initiation and in the damage development for thermal and mechanical loads. In a second step the developed material models are applied to a

RVE with a randomized fibre arrangement [44][45] to detect effects that may only occur in an irregular fibre architecture, see Figure 7. This supports and extends the findings about the micro scale phenomena obtained from the hexagonal RVE. The effective macroscopic properties due to micro damage are assumed to be affected less enough by the fibre arrangement for using the computational more efficient hexagonal RVE to derivation the equivalent stress state. Therefore, the randomized RVE is used for the phenomenological investigation but is not used in the multi scale computation.

In conjunction with the hypotheses, fatigue damage under thermal cycling is modelled based on the thermally induced stresses. As fibres and matrix are modelled explicitly material models are needed for both constituents. The fibres are assumed to behave linear elastic and transversal isotropic. The elastic and strength properties of the investigated IM7 fibre can be taken from [25] and are given in Table 1. Decreasing failure strains of the neat resin [26] indicate the reduction of plastic effects with decreasing temperatures. Therefore, the matrix is modelled fully elastic but nonlinear in the stress-strain-behaviour. The elastic properties and the strengths are given in Table 2. The nonlinearity in the stress-strain-behaviour can be captured by the formula

$$E = \frac{E_0}{1 + \eta |\varepsilon|} \varepsilon$$

used by Kästner et. al. [27][28] to model the elastic part of the constitutive behaviour of polypropylene. Herein, E_0 is the initial Young's modulus in the linear regime, E is the on the strain ε dependent effective Young's modulus and η is the parameter controlling the grade of nonlinearity. It is obtained by a regression processing using the test data from uniaxial tests, shown in Figure 8. To capture the difference in the compression and tension behaviour of the matrix material, an orthotropic formulation of the stiffness matrix is used. This allows to differentiate between compression and tension for each strain component and using the corresponding material parameters for tension or compression, respectively. For modelling failure initiation in the matrix under quasi-static loading, an invariant based stress failure criterion is used considering the effect of hydrostatic pressure on material failure [47]. After failure initiation elastic properties are reduced to approximately zero in order to assess the damage progression behaviour.

Figure 9 demonstrates the validity of the material model to predict the UD composite behaviour for quasi-static mechanical loading in transverse fibre direction at room temperature. The local stress distribution is exemplarily shown for the hexagonal RVE loaded under transverse tension in Figure 10. The figures show the distribution of the equivalent stress by von Mises (Figure 10(a)) and the distribution of the volumetric stress part (Figure 10(b)). As expected, the highest absolute stress values, especially for the volumetric stress part, occur at the fibre matrix interface. This is why the crack initiates at these positions, runs along the interface and then connects the interface damages forming a crack perpendicular to the loading direction, as shown in (Figure 10(c)).

325

330

335

340

345

350

For the micromechanical fatigue calculation, a fatigue model for the epoxy is needed which should consider mean stress effects and variable amplitudes. The Palmgren-Miner-rule [48][49] is appropriate for modelling damage accumulation and thereby considering variable loading amplitudes. If fatigue failure occurs for a material point, its elastic properties are reduced to approximately zero for considering load redistribution within the composite. The fibre-matrix-interface is assumed to play a key role in the damage and fatigue of FRP under thermal loads. The results of the single fibre tests described above will be used to create an interface model. Cohesive zones or cohesive element approaches already used for interface modelling [5][48][51][52] can be the basis. The novelty will be that the parameters of the cohesive model can be calibrated by single fibre test results and are not needed to be determined by reverse engineering using UD composite test data.

The micromechanical fatigue simulation results have to be validated against experimental data, too. For this purpose fatigue data of cyclically uniaxial loaded UD laminates are available in the literature [54][55][56] for the investigated composite. Herein, test data for axial and transverse tension and compression loads and also for in-plane shear are published and can be used as validation basis for corresponding simulations.

The RVEs shown in Figure 6 and Figure 7 together with the described material modelling approaches can be used to predict crack initiation and single crack growth

phenomena due to thermal cyclic loading as well as the resulting reduction of UD ply properties (Young's modulus and strength). These can be validated against the results of the thermo-cycling tests of UD laminates.

In addition to comparing the fatigue damage mechanisms under mechanical and thermal loads, the micromechanical model is also used to derive the mechanical stress state which is equivalent to the thermal cyclic loading. The main idea is sketched in Figure 12. Using the micromechanical model, curves of macroscopic damage against number of cycles are created for both, thermal and mechanical loading. For a given thermal load ΔT and a given number of thermal cycles N_{th} the degradation of the macroscopic material parameters of the UD laminate is determined. The equivalent mechanical stress state is this specific stress state σ which causes the same macroscopic damage at the same number of cycles ($N_{mech} = N_{th}$).

5. Macro scale – level of the cross-ply laminate

355

360

365

375

380

The macro scale looks at MD composites built up of several UD plies of different fibre orientation. In these composites, the micro and the macro effect are superimposed.

5.1 Fatigue phenomena in thermally cycled composites at the macro scale

Cross-ply laminates $[(0/90)_4]_s$ are thermally cycled, mechanically tested and investigated by microscopy in the same manner as the UD specimens described in the previous section. The experimental results for the cross-ply laminates published in [30] are summarized in the following: Matrix cracks initiate between 500 and 1000 thermal cycles in both 90° and 0° plies in the middle plies and in the outermost plies of the cross-ply laminate. The observed matrix crack density is too small to cause a significant reduction of the laminate's stiffness and strength. Nevertheless, these results support the assumption that the superposition of alternating thermally induced stresses on both, micro and macro scale is more severe regarding the fatigue of FRP at

cycling thermal loads than the micro scale effect acting alone. The results of the thermal cyclic experiments shall be used for validating the equivalent stress state approach in conjunction with the macromechanical fatigue modelling.

5.2 Macromechanical modelling of fatigue phenomena in FRP

385 Also for the fatigue modelling of MD laminates, FEM is used. For being able to capture the macromechanical effect, all single plies have to be modelled explicitly and the material modelling has to be done at the UD ply level. Again, the fatigue damage due to thermal cycling is assessed based on the thermally induced stresses. For modelling fatigue damage in UD composites an extended version [56] of the 390 progressive fatigue damage model by Shokrieh and Lessard [57][58] is suggested. This model can capture the mean stress effect and the gradual degradation of stiffness and strength with increasing number of cycles. In [56] the good prediction capability of the model for mechanical fatigue loads at different isothermal conditions is shown. Due to the homogenisation of UD plies, the macromechanical modelling is not applicable to calculate the crack density observed in thermally cycled MD laminates. 395 Arteiro et. al. [53] describe a RVE approach for investigating micro cracking under monotonic mechanical loads in UD plies which are embedded within MD laminates. The RVE consists of a single ply, which is micromechanically modelled, and adjacent plies considered as homogenous UD composites (ref. Figure 11). The same idea can 400 be used to numerically investigate micro crack development in a single UD ply embedded within a thermally cycled MD laminate. The results regarding crack initiation, crack pattern and crack density can be validated against the micro cracking experimentally observed. For the micromechanically modelled ply the material models of fibre and matrix are needed. For the homogenously modelled plies of the remaining laminate, the macromechanical material modelling approach for UD 405 composites can be used. One approach to consider the micro effect for these plies could be the scale transfer concept described in the next section.

6. Scale transfer by equivalent stress approach

In the case of thermal cyclic loading, for considering the thermally induced stresses at micro scale in spite of a macro scale modelling, the bottom-up approach sketched in Figure 2 in conjunction with the equivalent mechanical stress state described in the micro scale section is proposed. The framework in Figure 13 shows how the equivalent stress state connects the micro- and macromechanical modelling and how the information about the micro damage is passed into the macromechanical simulation. The micromechanical simulation is not performed simultaneously with the macromechanical simulation but is a pre-processing calculation step. Considering the constituents' material properties and the morphologic information, the hexagonal RVE is build up. With this RVE and using the described constituent (fibre, matrix, interface) material models, cyclic fatigue simulations for different numbers of cycles, each followed by quasi-static mechanical analyses, are run for different mechanical and thermal load levels. The cyclic simulations provide the micro damage due to a specific load and a specific number of cycles. In the followed quasi-static analysis, uniaxial mechanical loading is applied along and transverse to the fibre direction in tension and compression, and in shear as well, to obtain the residual composite properties (modulus and strength) after the cyclic loading. This step is the connection between the micro damage (matrix crack at micro level) and macro damage (property degradation of UD composite). It can be seen as translation of the micro damage into a macroscopic damage value which can be handled by the macromechanical model. For that, the micromechanical simulation results are used to construct the macro damage versus number of cycles diagrams (Figure 12) for the different thermal and mechanical load levels. From these diagrams, an analytical relationship between cyclic thermal loading and mechanical loading, both leading to the same macroscopic damage at the same number of cycles, is derived. The mechanical loading is called the equivalent mechanical stress state corresponding to a specific cyclic thermal load. After the derivation of this analytical relationship, the macromechanical simulation of thermo-mechanical loaded composites can be performed. To build up the macromechanical FE model, the UD ply properties, the geometry, the layup and the boundary conditions are needed. Also the mechanical and thermal loads, for which the fatigue calculation shall be performed, are applied. At each load increment of the macromechanical computation loop, the UD ply stresses due to the applied external

410

415

420

425

430

435

loads are calculated using the UD ply based macromechanical material model. Without considering the micro effect, after each load cycle, these macro stresses would alone be assessed by the presented UD ply based macromechanical fatigue damage model. For considering the micro effect, after each load cycle, the equivalent stress state for the given cyclic thermal load is calculated form the analytical relationship created by the micromechanical pre-processing. This equivalent stress state is added to the macro stresses and together they are assessed by the macromechanical fatigue damage model. If the damage model predicts static or fatigue failure, the ply properties are degraded and the next increment and load cycle is calculated using these new properties.

445

450

455

460

465

470

As the equivalent stress state is obtained by an analytical formula during the macromechanical simulation, the computation time is not higher than for a macromechanical simulation without considering the micro effect. This makes the calculation of large structures possible. It could be argued that the computational effort is shifted to the micromechanical pre-processing. But this has to be done only once for a chosen material system and can be limited to thermal loads which are relevant to the application of interest. Once the analytical relationship for the equivalent stress state is created, any number of large structure simulations can be performed without additional calculation time due to considering thermal micro effects. Nevertheless, the described approach has to be evaluated and validated. This can be done by comparing the simulation results with the results of thermal cycling tests of MD laminates.

7. Summary and outlook of experimental and numerical approach

This paper presents an experimental and numerical approach for investigating thermally cycled FRP which contributes to the understanding of the damage mechanisms and increase the predictability of the fatigue damage at thermal loading conditions. This includes clearly focused experiments at the different material scales for separating the different effects acting at these scales. In parallel, numerical models are proposed for each scale based on the experimental findings. The models also

complement the knowledge gained from experiments and create a basis for a numerical prediction capacity.

475

480

485

490

495

500

The central hypothesis leading the test and model design is that different thermal expansions of fibre and matrix lead to intra-ply stresses at the micro level and interply stresses at the macro level. They are responsible for damage and fatigue of cyclic thermal loaded FRP. Tests of the presented research have shown that the interaction of stresses at both levels (e.g. in cross-ply laminates) leads to more severe damage than the inter-ply stresses (e.g. in UD laminates) acting alone. A further hypothesis is the weakening of the fibre-matrix-interface due to thermal cycling. To proof this hypothesis, experimental studies based on the already existing single fibre pull-out and peel-off devices are suggested. A new sample design is proposed to emulate the stress state around a fibre within a thermal loaded real composite material in these tests. It is expected that the interfacial shear strength obtained from pull-out test decrease with the number of cycles. As for peel-off the contact length is much larger then with the pull-out samples (some millimetres instead of few dozens of micrometres), potentially thermally induced phenomena may lead to a faster degradation with a lower number of thermal cycles. From single fibre peel-off tests, the energy required for fibre-matrix separation is obtained. This energy as a function of cycling numbers can be compared with the function of the interface shear strength values from the pull-out tests.

Numerical modelling accompanies the test campaign, because simulations support the explanation of phenomena observed in experiments. In the presented research, it is proposed to use micromechanical fatigue modelling based on RVE approach to explain the differing experimentally observed crack propagation in FRP obtained for mechanical and thermal fatigue loading. This proposal is based on the assumption that these differences result from the different inner stress states caused by mechanical and thermal loads. These stress states can be analysed by the micromechanical computations using material models for fibre and matrix taking into account material nonlinearity, damage at monotonic loading and fatigue damage behaviour at different mean stresses and temperatures. The single fibre experiments offer the opportunity to calibrate interface models by test data at micro level avoiding a calibration by reverse

engineering using macroscopic UD ply test data. Thanks to the comprehensive test program the obtained interface parameters can be defined dependent on the number of thermal cycles and the weakening of the interface due to thermal cycling can be captured in the micromechanical model. Comparing the simulation results for thermal and mechanical loading the differing experimentally observed crack patterns can be explained. By homogenisation, also the impact on the effective composite properties can be compared for both loading types. From this, an equivalent mechanical stress, leading at the same cycle number to the same macroscopic damage as a specific thermal load, can be derived. This way, the on micro scale induced thermal stresses are, together with the impact of other considered nonlinearities, incorporated in the equivalent mechanical stress and can be transferred to the macro scale (bottom-up). This can be done by analytical expressions relating the given thermal fatigue load to the equivalent mechanical stress. Then, during the macromechanical fatigue analysis, the equivalent mechanical stress can easily be computed and be assessed by the proposed macroscopic progressive fatigue damage model. This way a simulation method will be available considering the micro effect of thermal cycling even in homogenous modelled UD composites and which also captures nonlinearities. After an evaluation and validation of this method, it can be used for comparing simulation results with and without considering the micro effects providing a possibility to analyse the relevance of the micro effect for fatigue of FRP at thermal cyclic loads. This analysis cannot be done experimentally.

Funding

505

510

515

520

This work was supported by the German Research Foundation, DFG [project number 379482657].

Abbreviations

CcH2 cryo-compressed hydrogen

530 CFRP carbon fibre reinforced plastics

FEM finite element method

FRP fibre reinforced plastics

LN2 liquid nitrogen

MD multidirectional

535 RVE representative volume element

UD unidirectional

Formula symbols

E Young's modulus

 E_0 Initial Young's modulus

 F_{max} aximal pull-out force

l_e embedding length

Number of cycles

N_{th} Number of thermal cycles

 N_{mech} Number of mechanical cycles

545 R_f fibre radius

 ε strain

 η Nonlinearity parameter

 σ stress

τ shear strength

550 Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

References

- [1] Kircher O, Greim G, Burtscher J, Brunner T. Validation of cryo-compressed
 Hydrogen Storage (CCH2)- A Probalistic Approach. 4th Int Conf Hydrog Saf.
 San Francisco, California; 2011
 - [2] Grammatikos SA, Jones RG, Evernden M, Correia JR. Thermal cycling effects on the durability of a pultruded GFRP material for off-shore civil engineering structures. Compos Struct; 2016;153:297–310
- 560 [3] Hegde SR, Hojjati M. Thermally induced microcracks and mechanical property of composite honeycomb sandwich structure: Experiment and finite element analysis. J Sandw Struct Mater. 2018;
 - [4] Hartwig G, Knaak S. Fibre-epoxy composites at low temperatures. Cryogenics;1984;24(11):639–647.
- 565 [5] Vaughan TJ, McCarthy CT. Micromechanical modelling of the transverse damage behaviour in fibre reinforced composites. Compos Sci Technol.; 2011;71(3):388–96.
 - [6] Asp LE, Berglund LA, Talreja R. Prediction of matrix-iniated transverse failure in polymer composites. Compos Sci Technol; 1996;56:1089–97.
- 570 [7] Lafarie-Frenot MC, Hénaff-Gardin C, Gamby D. Matrix cracking induced by cyclic ply stresses in composite laminates. Compos Sci Technol;2001;61(15):2327–36.
- [8] Timmerman JF, Tillman MS, Hayes BS, Seferis JC. Matrix and Fibre influences on the cryogenic microcracking of carbon fiber / epoxy composites. Compos Part A: Appl Sci Manuf. 2002;33:323–329.
 - [9] Brown TL. The Effect of Long-Term Cycling on the Microcracking Behaviour and Dimensional Stability of Composite Materials. Virginia Polytechnic Institute and State University, PhD Thesis; 1997.
- [10] Christian M. The elastic Stress Field arising in the Single Fiber Pull-out Test.

 Composites science and technology. 1994 (50): 393-405

- [11] Hampe A, Kalinka G, Meretz S, Schulz E. An advanced equipment for single-fibre pull-out test designed to monitor the fracture process. Composites 1995 (26): 40-46.
- [12] Godara A, Gorbatikh L, Kalinka G, Warrier A, Rochez O, Mezzo L, u. a. Interfacial shear strength of a glass fiber/epoxy bonding in composites modified with carbon nanotubes. Compos Sci Technol. Elsevier Ltd; 2010;70(9):1346-52.
 - [13] Hodzic A, Stachurski ZH, Kim JK. An Analysis of Microdroplet Test: Effects of Specimen Geometry, Matrix Type, Fibre Treatment and Water Ageing. Polym Polym Compos. 2001;9(8):499–508.
- 590 [14] Hodzic A, Kalyanasundaram S, Lowe A, Stachurski ZH. The microdroplet test: experimental and finite element analysis of the dependance of failure mode on droplet shape. Compos Interfaces. 1999;6(4):375–89.
 - [15] Schultz J, Nardin M. Some Physico-Chemical Aspects of the Fibre-Matrix Interphase in Composite Materials. J Adhes. 1994;45(1–4):59–71.
- 595 [16] Nardin M, Schultz J. Relationship between fibre-matrix adhesion and the interfacial shear strength in polymer-based composites. Compos Interfaces ISSN. 1993;1(2):177–92.
- [17] Zhandarov S, Mäder E, Kalinka G, Scheffler C, Poitzsch C, Fliescher S.
 Investigation of interfacial strength parameters in polymer matrix composites:
 Compatibility and reproducibility. Advanced Industrial and Engineering Polymer Research 2018 (1): 82-92
 - [18] Sahin M, Schlögl S, Kalinka G, Wang J, Kaynak B, Mühlbacher I, Ziegler W, Kern W, Grützmacher H. Tailoring the interfaces in glass fiber-reinforced photopolymer composites. Polymer 2018 (14): 221-231
- 605 [19] Henaff-Gardin C, Lafarie-Frenot MC. Specificity of matrix cracking development in CFRP laminates under mechanical or thermal loadings. Int J Fatigue.2002;24:171–177.

[20] Adams DS, Bowles DE, Herakovich CT. Thermally Induced Transverse Cracking in Graphite-Epoxy Cross-Ply Laminates. J Reinf Plast Compos. 1986;5:152–69.

610

615

- [21] Lafarie-Frenot MC, Ho NQ. Influence of free edge intralaminar stresses on damage process in CFRP laminates under thermal cycling conditions. Compos Sci Technol. 2006;66(10):1354–65
- [22] Kim RY, Crasto AS, Schoeppner GA. Dimensional stability of composite in a space thermal environment. Compos Sci Technol. 2000;60:2601–8.
 - [23] Fahmy AA, Cunningham TG. Investigation of Thermal Fatigue in Fiber Composite Materials. Technical Report, National Aeronautics and Space Administration, Washington, D.C.; 1976
- [24] Hancox N. Thermal effects on polymer matrix composites: Part 1. Thermal cycling. Materials & Design. 1998;19(3):85–91.
 - [25] Kaddour AS, Hinton MJ. Input data for test cases used in benchmarking triaxial failure theories of composites. J Compos Mater. 2012;46(19–20):2295–312.
 - [26] Lin S, Jia X, Sun H, Sun H, Hui D, Yang X. Thermo-mechanical properties of filament wound CFRP vessel under hydraulic and atmospheric fatigue cycling. Compos Part B Eng; 2013;46:227–33.
 - [27] Kästner M, Haasemann G, Ulbricht V. Multiscale XFEM-modelling and simulation of the inelastic material behaviour of textile-reinforced polymers. Int J Numer Methods Eng. 2011;86:477–98
- [28] Kästner M, Obst M, Brummund J, Ulbricht V. Influence of the nonlinear matrix material behaviour on the effective properties of textile-reinforced thermoplastics. 2009;342:341–2.
 - [29] Dutta PK, Hui D. Low-temperature and freeze-thaw durability of thick composites. Compos Part B Eng. 1996;27(3–4):371–8.
- [30] Lüders C, Sinapius M. Fatigue of fibre-reinforced plastics due to cryogenic thermal cycling. J Compos Mater. 2019;

- [31] Chamis CC, Ginty. CA. Fiber composite structural durability and damage tolerance: simplified predictive methods. In: 2nd Symposium on Composite Materials: Fatigue and Fracture. Cincinnati, Ohia; 1987.
- [32] Shimokawa T, Katoh H, Hamaguchi Y, Sanbongi S, Mizuno H, Nakamura H, u.

 a. Effect of thermal cycling on microcking and strength degradation of hightemperature polymer composite materials for use in next-genaration SST
 structures. J Compos Mater. 2002;36(07):885–95.
 - [33] Kang SG, Kim MG, Kim CG, Lee JR, Kong CW. Thermo elastic analysis of a type 3 cryogenic tank considering curing temperature and autofrettage pressure. J Reinf Plast Compos. 2008;27(5):459–72.

- [34] Grogan DM, Ó Brádaigh CM, McGarry JP, Leen SB. Damage and permeability in tape-laid thermoplastic composite cryogenic tanks. Compos Part A Appl Sci Manuf; 2015;78:390–402.
- [35] Henaff-Gardin C, Lafarie-Frenot MC, Gamby D. Doubly periodic matrix cracking in composite laminates Part 2: Thermal biaxial loading. Compos Struct. 1997;36(96):131–40.
 - [36] Park CH, McManus HL. Thermally induced damage in composite laminates: Predictive methodology and experimental investigation. Compos Sci Technol. 1996;56(10):1209–19
- 655 [37] McManus HL, Bowles DE, Tompkins SS. Prediction of Thermal Cycling Induced Matrix Cracking. J Reinf Plast Compos. 1996;15.
 - [38] Shah AR, Murthy PLN, Chamis C. Effect of Cyclic Thermo-Mechanical Load on Fatigue Reliability in Polymer Matrix Composites. In: 36th Structure, Structural Dynamics and Materials Conference. New Orleans, Louisiana; 1995
- [39] Shah AR, Chamis CC. Effect of Cyclic Thermal Loads on Fatigue Reliability in Polymer Matrix Composites. In: 37th Structure, Structural Dynamics and Materials Conference. 1996

[40] Huang C, Ren M, Li T, Chang X, Cong J, Lei Y. Trans-scale Modelling Framework for Failure Analysis of Cryogenic Composite Tanks. Compos Part B Eng; 2015;85:41–9.

665

- [41] Ren M, Zhang X, Huang C, Wang B, Li T. An integrated macro/micro-scale approach for in situ evaluation of matrix cracking in the polymer matrix of cryogenic composite tanks. Compos Struct; 2019
- [42] Wang L, Zheng C, Wei S, Wei Z. Micromechanics-based progressive failure analysis of carbon fiber/epoxy composite vessel under combined internal pressure and thermomechanical loading. Compos Part B Eng; 2016;89:77–84.
 - [43] Alimuddin MA, Piggott MR. Fracture Toughness of Fiber-Polymer Interfaces Estimated From Single Fiber Peel Tests. Polym Compos. 1999;20(5):655–63.
- [44] Krause D. A physically based micromechanical approach to model damage initiation and evolution of fiber reinforced polymers under fatigue loading conditions. Compos Part B Eng; 2016;87:176–95.
 - [45] Krause D. Mikromechanik des Ermüdungsverhaltens polymerer Verbundwerkstoff. TU Braunschweig; PhD Thesis. 2016.
- [46] Kästner M. Skalenübergreifende Modellierung und Simulation des mechanischen
 Verhaltens von textilverstärktem Polypropylen unter Nutzung der XFEM.
 Technische Universität Dresden; PhD Thesis. 2009.
 - [47] Ha SK, Jin KK, Huang Y. Micro-Mechanics of Failure (MMF) for Continuous Fiber Reinforced Composites. J Compos Mater. 2008;42(18):1873–95.
- [48] Palmgren A. Die Lebensdauer von Kugellagern. Zeitschrift des Vereines Dtsch Ingenieure. 1924;68(14):339–41.
 - [49] Miner MA. Cumulative Damage in fatigue. J Appl Mech. 1945;12(3):159-64.
 - [50] Tan W, Naya F, Yang L, Chang T, Falzon BG, Zhan L, u. a. The role of interfacial properties on the intralaminar and interlaminar damage behaviour of unidirectional composite laminates: Experimental characterization and multiscale modelling. Compos Part B Eng;2017;138(October 2017):206–21.

- [51] Vaughan TJ, McCarthy CT. A micromechanical study on the effect of intra-ply properties on transverse shear fracture in fibre reinforced composites. Compos Part A Appl Sci Manuf. Elsevier Ltd; 2011;42(9):1217–28.
- [52] Sun Q, Meng Z, Zhou G, Lin S, Kang H, Keten S. Multi-scale computational analysis of unidirectional carbon fi ber reinforced polymer composites under various loading conditions. Compos Struct; 2018;196(December 2017):30–43.
 - [53] Arteiro A, Catalanotti G, Melro AR, Linde P, Camanho PP. Micro-mechanical analysis of the in situ effect in polymer composite laminates. Compos Struct [Internet]. Elsevier Ltd; 2014;116:827–40.
- 700 [54] Hamid ZM binti A, Hohe J, Gall M, Fliegener S, Gumbsch P. Fatigue Damage and Degradation Model for Carbon Fibre Reinforced Polymer Materials. Proc Appl Math Mech. 2017;17(1):259–60.
 - [55] Hohe J, Gall M, Gauch H, Fliegener S, Hamid ZM binti A. A Material Model for Prediction of Fatigue Damage and Degradation of CFRP Materials. In: 21st Symposium on Composites, Key Engineering Materials. Trans Tech Publications; 2017. S. 740–4.
 - [56] Lüders C, Krause D, Kreikemeier J. Fatigue damage model for CFRP at different temperatures considering stress ratio effects. J Compos Mater. 2018.
- [57] Shokrieh MM, Lessard LB. Progressive Fatigue Damage Modeling of Composite
 Materials, Part I: Modeling. J Compos Mater. 2000;34:1081–116.
 - [58] Shokrieh MM, Lessard LB. Progressive Fatigue Damage Modeling of Composite Materials, Part II: Material Characterization and Model Verification. J Compos Mater. 2000;34(13):1081–116.

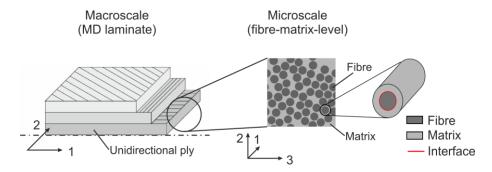


Figure 1 Considered material scales

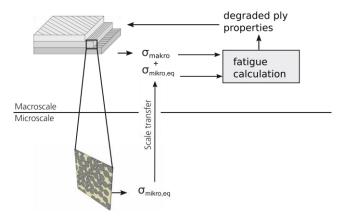


Figure 2 Method of bottom-up approach

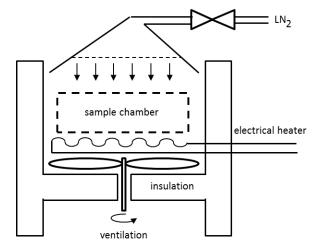


Figure 3 sketch of the temperature cycling chamber for small scale samples



Figure 4 Sketch of a single fibre pull-out sample embedded between platelets of UD composite material



Figure 5 Sketch of a single fibre peel-off sample: a single fibre is glued on the top side of a UD composite material

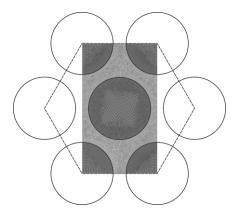


Figure 6 Representative volume element with hexagonal fibre arrangement with carbon fibres (dark grey) and epoxy resin (light grey)

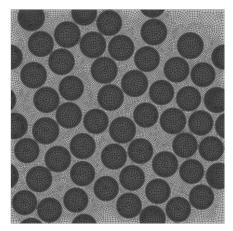
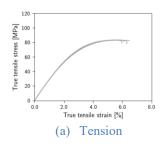


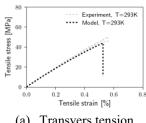
Figure 7 Representative volume element with randomised fibre arrangement with 740 carbon fibres (dark grey) and epoxy resin (light grey)



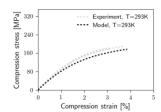
250 200 150 100 10.0 20.0 True compres 30.0 40.0 ion strain [%] (b) Compression

745

Figure 8 Uniaxial test data of pure resin



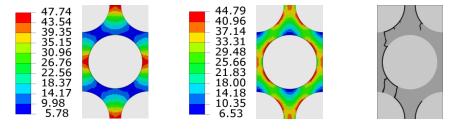
(a) Transvers tension



(b) Transverse compression

Figure 9 Comparison of micromechanical prediction of the mechanical UD composite behaviour at room temperature and experimental results for (a) transverse tension, (b) transverse compression

770



(a) von Mises stresses [MPa] (b) hydrostatic stresses [MPa] (c) matrix crack

Figure 10 Local stress state in UD composite under transverse tension: (a) equivalent von Mises stresses, (b) hydrostatic stresses (positive – tension, negative – compression) and (c) the matrix cracked formed (damage areas are coloured black);

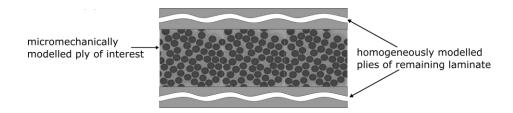


Figure 11 Adoption of RVE approach by Arteiro et. al. [53]: RVE consisting of a micromechanically modelled ply of interest and homogenously modelled plies of the remaining laminate

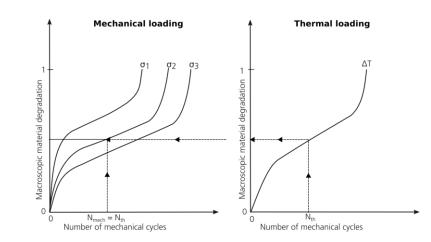


Figure 12 Equivalent mechanical stress state

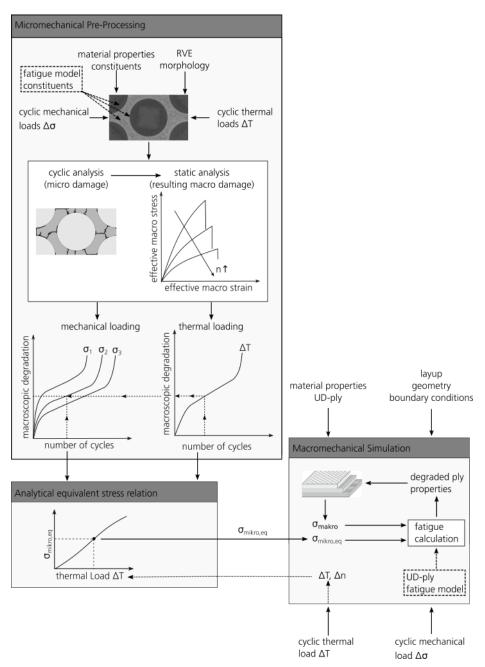


Figure 13 Flowchart of multi-scale approach using equivalent stress assumption

Table 1 Elastic and strength properties of IM7 fibre

E_{11}	E_{22}	E_{33}	G_{12}	G_{13}	G_{23}	v_{12}	v_{12}	v_{23}	X_{l}	X_{c}
[GPa]	[GPa]	[GPa]	[GPa]	[GPa]	[GPa]	[-]	[-]	[-]	[GPa]	[GPa]
276	19	19	27	27	7	0.2	0.2	0.357	5.18	3.2

Table 2 Elastic and strength properties of epoxy resin

E	G	ν	X_{l}	X_{c}
[MPa]	[MPa]	[-]	[MPa]	[MPa]
3110	1114	0.395	82	262